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IS 4105 (1988): Styrene (Vinyl Benzene) [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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Indian Standard

SPECIFICATION FOR STYRENE (VINYL BENZENE)

(First Revision)

UDC 547·538·141

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 7

February 1989

Indian Standard

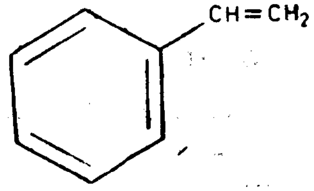
SPECIFICATION FOR STYRENE (VINYL BENZENE)

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 20 May 1988, after the draft finalized by the Organic Chemicals (Misc) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Styrene ($C_6H_5-CH=CH_2$) is used for the manufacture of styrene-butadiene rubber (SBR) polystyrene and for copolymerization products with other monomers to obtain synthetic resin binders and adhesives. It is also used in the packaging industry and in the production of toys, housewares and appliances. Styrene-divinyl benzene copolymer is used in the preparation of both anion and cation exchange resins. It is also known as phenyl ethylene or vinyl benzene. It is represented by the following structural formula:



Styrene (Vinyl benzene)
(Molecular mass 104·15)

0.3 The material undergoes polymerization during shipment or in storage due to the catalytic effect of heat and light. The commercial material is, therefore, stabilized with sulphur or other inhibitors like *p*-tertiary butyl catechol in small concentrations.

0.4 This standard was first published in 1967. It was decided to revise it in view of the improved quality of the material being made available in the country. In the revised version, the requirement of refractive index and relative density have been included, limits of impurity stipulated and the methods of test for colour and aldehyde content modified.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values (*revised*).

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for the material commercially known as styrene (vinyl benzene).

2. GRADES

2.1 The material shall be of two grades, namely, Grade 1 and Grade 2.

a) *Grade* 1 — for plastic industry, and

b) *Grade* 2 — for rubber industry.

3. REQUIREMENTS

3.1 Description — The material shall be clear liquid free from sediment, suspended matter and undissolved water at 27°C.

3.2 The material shall also comply with the requirements given in Table 1.

4. PACKING AND MARKING

4.1 Packing

4.1.1 Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in internally epoxy coated steel drums painted white externally.

4.1.2 All containers shall be dry, clean and free from substances soluble in styrene and leak-proof.

4.1.3 The containers shall be securely closed, protected from light and heat and shall be stored in a cool place.

TABLE 1 REQUIREMENTS FOR STYRENE (VINYL BENZENE)

(*Clause* 3.2)

SL No.	CHARACTERISTIC	REQUIREMENT FOR		METHOD OF TEST, REF. TO	
		Grade 1	Grade 2	Indian Standard	Cl No. of Appendix A
(1)	(2)	(3)	(4)	(5)	(6)
i)	Colour	To pass the test	To pass the test	—	A-1
ii)	Relative density at 27/27°C	0·901 5 to 0·903 0	0·901 5 to 0·903 0	—	A-2
iii)	Refractive index at 27°C, *Min*	1·542 6	1·542 4	—	A-3
iv)	Freezing point °C, *Min*	−30·79	−30·95	—	A-4
v)	Assay, percent by mass, *Min*	99·60	99·20	—	A-5
vi)	Aldehyde, percent by mass (as benzaldehyde), *Max*	0·02	0·05	—	A-6
vii)	Peroxides, percent by mass (as H_2O_2), *Max*	0·01	0·01	—	A-7
viii)	Sulphur, percent by mass, *Max*	0·003	0·005	IS : 1448 (P : 34)-1979*	—
ix)	Chlorides, percent by mass, *Max*	0·005	0·01	—	A-8
x)	Polymer content, ppm, *Max*	10	50	—	A-9
xi)	Solubility of polymer in toluene	Wholly soluble	Wholly soluble	—	A-10
xii)	Inhibitor content (*p*-tertiary butyl catechol), ppm, *Min*†	50	50	—	A-11
xiii)	Ethyl benzene, percent by mass, *Max*	0·35	0·50	—	A-12
xiv)	Alpha methyl styrene, percent by mass, *Max*	0·05	0·30	—	A-12

*Methods of test for petroleum and its products: P:34 Determination of sulphur in petroleum products (lamp method) (*second revision*).

†Recommended for one month's safe storage under Indian climatic conditions. May be increased or decreased by mutual agreement between the purchaser and the supplier.

4.1.4 All containers for storage and transport of the material, since classified as flammable and dangerous goods, shall in addition, comply with the requirements of the latest issue of Red Tarrif and the requirements as laid down from time to time by the Chief Inspector of Explosives, Government of India, for packing, storage and transit of flammable liquids and the Board of Trade Regulations as applicable thereon for transport by steamers.

4.1.5 Except when they are opened for the purpose of cleaning and rendering them free from styrene vapour, all empty tanks or other containers shall be kept securely closed unless they have been thoroughly cleaned and freed from styrene vapour.

4.2 Marking

4.2.1 Each container shall have a caution label 'FLAMMABLE' together with the corresponding symbol for labelling of dangerous goods in accordance to IS : 1260 (Part 1)-1973*.

NOTE — Necessary safeguard against the risk arising from storage and handling of large volumes of flammable liquids shall be provided and all due precautions shall be taken at all times to prevent accidents by fire or explosion.

4.2.2 Each container shall bear legibly and indelibly the following information:

a) Name and grade of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Batch number and date of manufacture;

d) Tare, net and gross mass; and

e) Inhibitor content.

4.2.3 The container may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard

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*Specification for pictorial markings for handling and labelling of goods: Part 1 Dangerous goods (*first revision*).

marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 The procedure for sampling and criteria for conformity of the material shall be as prescribed in Appendix B.

6. TEST METHODS

6.1 Tests shall be carried out according to the methods prescribed in IS : 1448 (P : 34)-1979* appendices to this standard. References to the relevant method of IS : 1448 (P : 34)-1979* and appendices are given in Table 1.

6.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Methods of test for petroleum and its products: P:34 Determination of sulphur in petroleum products (lamp method) (*second revision*).

†Specification for water for general laboratory use (*second revision*).

APPENDIX A

(*Table* 1)

METHODS OF TEST FOR STYRENE (VINYL BENZENE)

A-1. DETERMINATION OF COLOUR

A-1.0 Outline of the Method — Colour of the material is compared to standard colour under identical conditions.

A-1.1 For Grade 1

A-1.1.1 *Reagents*

A-1.1.1.1 *Potassium bichromate solution (0·002 5 N)* — Dissolve 0·122 6 g of analytical grade potassium bichromate conforming to IS : 250-1964*, dried at 120 to 140°C for about three hours, in water and dilute to 1 000 ml.

A-1.1.1.2 *Sulphuric acid* — 1 percent (*m/v*).

A-1.1.2 *Procedure* — In a 100-ml Nessler tube, take 2·6 ml of freshly prepared 0·002 5 N potassium bichromate solution and dilute it to 100-ml mark with 1 percent sulphuric acid. In a similar 100-ml Nessler tube, take a sample of styrene up to 100-ml mark and compare the colour on white tile viewing from the top.

A-1.1.3 The material shall be taken to have passed the test if the solution is not darker than the diluted potassium bichromate solution.

A-1.2 For Grade 2

A-1.2.1 Reagents

A-1.2.1.1 *Potassium bichromate solution* — 0·002 5 N.

A-1.2.1.2 *Sulphuric acid* — 1 percent (*m/v*).

A-1.2.2 *Procedure* — In a 100-ml Nessler tube, take 3·7 ml of freshly prepared 0·002 5 N potassium bichromate solution and dilute it to 100-ml mark with 1 percent sulphuric acid. In a similar 100-ml Nessler tube, take a sample of styrene up to 100-ml mark and compare the colour on a white tile viewing from the top.

A-1.2.3 The material shall be taken to have passed the test if the solution is not darker than the diluted potassium bichromate solution.

A-2. DETERMINATION OF RELATIVE DENSITY

A-2.0 Outline of the Method — In this method, masses of equal volumes of the material and water at the same temperature are compared using relative density bottle.

A-2.1 Apparatus

A-2.1.1 Relative Density Bottle — 25 ml capacity.

A-2.1.2 *Water-Bath* — maintained at 27·0 ± 0·2°C.

A-2.1.3 *Thermometer* — Any convenient thermometer of suitable range with 0·1 or 0·2 deg sub-divisions.

A-2.2 Procedure — Clean and dry the relative density bottle, weigh and then fill with recently boiled and cooled water (*see* IS : 1070-1977*) at 27°C. Fill to overflowing by holding the relative density bottle on its side in such a manner as to prevent entrapment of air bubbles. Insert the stopper and immerse in a water-bath maintained at 27 ± 0·2°C. Keep the entire bulb

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*Specification for potassium bichromate, technical and analytical reagent (*revised*).

*Specification for water for general laboratory use (*second revision*).

completely covered with water and hold at that temperature for 30 minutes. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry, cool and weigh. Calculate the mass of water. Again clean and dry the relative density bottle. Using the material under test, proceed exactly as in the case of water and weigh the bottle with the material.

A-2.3 Calculation

$$\text{Relative density at } 27/27°\text{C} = \frac{A - B}{C - B}$$

where

A = mass in g of the relative density bottle filled with the material,

B = mass in g of the clean and dry relative density bottle, and

C = mass in g of the relative density bottle filled with water.

A-3. DETERMINATION OF REFRACTIVE INDEX

A-3.0 General — For the purpose of this determination, the refractive index of a material is taken as the ratio of the sine of the angle of incidence to the sine of the angle of refraction when a ray of light of wavelength 5 893 A° (the mean of the D lines of sodium) passes from air into the material.

The notation is refractive index $n \frac{t}{D}$, t being the temperature (°C) at which the determination is made.

A-3.1 Procedure — Determine the refractive index in a standard instrument* employing the principle of the critical angle, using diffused daylight or any convenient artificial light as illuminant. Maintain the prisms at the specified temperature and allow the material to stay on the prism surface for a couple of minutes to attain the required temperature. Take a second reading after the laps of a few minutes.

A-3.1.1 Since moisture in the air may condense on the cooled prisms, great care shall be exercised when determining refractive indices during hot, humid weather. Occasionally, the instrument should be checked by means of the quartz plate that accompanies it, using monobromonaphthalene, or if such a plate is not available, by means of distilled water at 30°C. The refractive index of distilled water at 30°C is 1·332 200.

A-3.1.2 If, for any reason, the refractive index cannot be determined at the specified temperature, apply the correction factor of 0·000 38 per degree Celsius if a factor is not so specified. If the refractive index is determined at a temperature above the specified temperature, add the appropriate correction; if determined below the specified temperature, subtract the appropriate correction.

A-3.1.3 Report the refractive index at 27°C as a number correct to four decimal places.

A-4. DETERMINATION OF FREEZING POINT

A-4.0 Outline of the Method — The freezing point of the material which is a measure of its purity, is determined in equilibrium with air atmospheric pressure. The presence of small amounts of impurities causes of depression of the freezing point which is proportional to the molar concentration of the contaminating substances. The freezing point is determined from the freezing curve — a graph of temperature versus time readings obtained when a stirred sample of the styrene is gradually cooled and frozen in a sample tube surrounded by an air bath refrigerated with dry ice. The measurements are made directly on sample without preliminary purification as small amounts of polymer do not affect the results because of low molar concentration. Any water present is frozen out of the sample and does not affect the results.

A-4.1 The method is applicable to samples in which the major impurity is ethyl benzene.

A-4.2 Apparatus

A-4.2.1 *Thermometer* — specially desiged to conform to the following specifications:

Liquid	: Mercury
Filling above mercury	: Nitrogen gas
Range and subdivision	: −33·02 to −29·98°C in 0·02 intervals with auxiliary scales from −0·2 to +0·2°C.
Total length	: 405 to 410 mm
Immersion	: Total
Stem	: Plain front, enamel back, suitable thermometer tubing, diameter 6 to 7 mm
Bulb	: Of suitable thermometric glass; diameter 6 to 7 mm but not greater than that of stem, length 50 to 60 mm
Distance from bottom of bulb to −33 degree mark	: 130 to 135 mm

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*A suitable type of instrument for this purpose is the Abbe Refractometer.

Distance from top of the thermometer to −30 degree mark	125 to 135 mm
Length of unchanged capillary between the enlargement and the graduation next below	Not less than 15 mm
Length of unchanged capillary above or below the 0° point	Not less than 15 mm
Expension chambers	To permit heating to at least 50°C
Top finish	Plain
Graduation	All lines, figures and letters to be clear cut and distinct. The graduation marks to be fine, straight, of uniform width and perpendicular to the axis of the thermometer. Each degree and the tenth degree line to be longer than the intermediate ones. Graduation to be numbered in full at each degree mark and in decimals at other multiples of 0·2°.
Special marking	The manufactures' name or trade-mark, a serial number and the words 'total immersion' shall be etched on the stem.
Scale error tolerance	The error at any point on the scale shall not exceed 0·1°C.

NOTE — The accuracy attainable with mercury-in-glass thermometers, based on performance, when all precautions are taken, for measurements of the kind for which these thermometers were designed, is from 0·01 to 0·03°C, and calibration corrections are stated to the nearest 0·01. Subdivision in 0·02 intervals, therefore, serves principally for the purpose of facilitating readings. This thermometer shall be compared to a standard thermometer of the same type certified by the National Physical Laboratory, New Delhi and periodically checked for its accuracy by ice point determination or against bromobenzene standard (*see* **A-4.7**) or both.

A-4.2.2 *Freezing Point Apparatus* — It shall be of the dimensions and shape as given in Fig. 1, made of Type 1 glass conforming to IS : 2303-1963*.

A-4.2.3 *Bath Thermometer* — for reading the temperature of the cryostat bath. It should read as low as −50°C and be graduated in 1° subdivisions.

A-4.3 Reagents

A-4.3.1 *Freezing Mixture* — A mixture of equal parts by volume of acetone and carbon tetrachloride for filling the Dewar flask.

A-4.3.2 *Carbon Dioxide, Solid* (*Dry Ice*)

A-4.3.3 Bromobenzene, extra pure. This should be kept in a dark bottle away from direct sunlight.

A-4.4 *Sample Size and Precautions for Storage* — A sample of 20 to 25 ml is sufficient for duplicate determination. Samples should be kept refrigerated or inhibited with a minimum of 10 ppm of *p*-tertiary butyl catechol to prevent polymerization.

A-4.5 *Procedure*

4.5.1 Use a sample of approximately 10 ml so that the top of the thermometer bulb will be immersed 2 cm when the bottom of the bulb is placed 1 cm above the bottom of the sample tube. Fill the Dewar flask with the acetone-carbon tetrachloride mixture previously cooled to about −42°C (by the addition of dry ice) to a point at least 1 cm above the level of the styrene in the freezing cell. Maintain the bath temperature between −44 and −48°C during the determination, by cautiously adding dry ice, a small piece at a time. Make sure that the bath does not foam excessively and that the carbon dioxide vapour is not allowed to contaminate the sample.

A-4.5.2 Adjust the stirrer to 100 to 150 strokes per minute and stir continuously throughout the determination. Be sure that the coil of the stirrer reaches the bottom of the sample tube and does not emerge from the liquid while in operation. Record the temperature to the nearest 0·01°C at 30-second intervals and plot the readings of temperature versus time on graph paper (Temperature-time data may be plotted directly at the time of observation upon graph paper prepared in advance). The freezing point of the sample, uncorrected, is the point of intersection of the liquid cooling curve of the temperature versus time graph with the freezing portion of the graph extrapolated back over the region of supercooling.

A-4.6 Calculation

A-4.6.1 The uncorrected freezing point as obtained by extrapolation (*see* **A-4.5.2**) must be corrected by adding to it, algebraically, the following quantities:

a) The calibration correction obtained by comparison to the standard thermometer;

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*Method of grading glass for alkalinity.

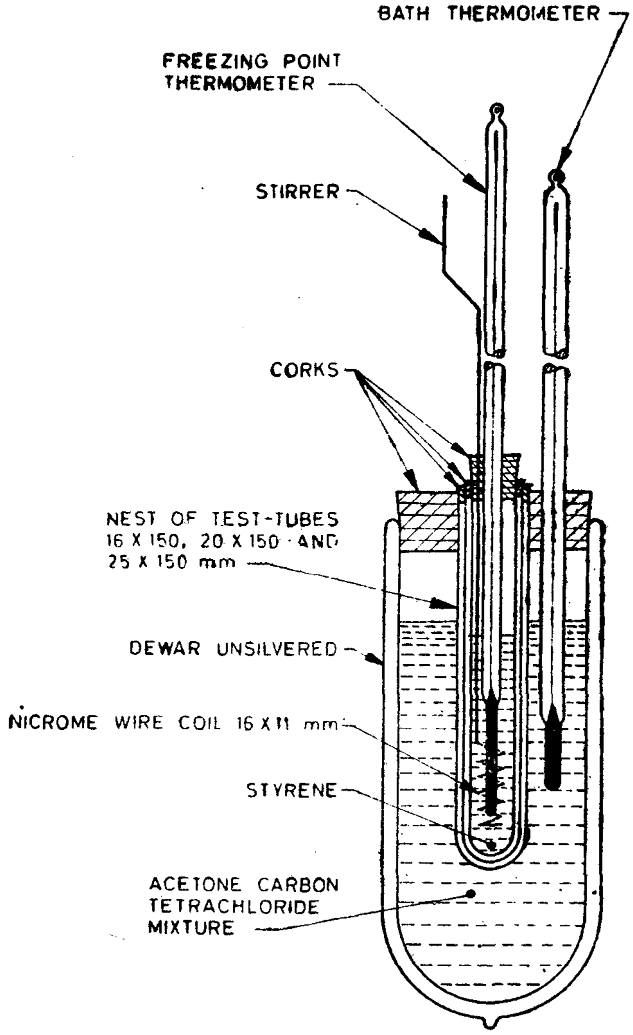

BATH THERMOMETER
FREEZING POINT THERMOMETER
STIRRER
CORKS
NEST OF TEST-TUBES 16 X 150, 20 X 150 AND 25 X 150 mm
DEWAR UNSILVERED
NICROME WIRE COIL 16 X 11 mm
STYRENE
ACETONE CARBON TETRACHLORIDE MIXTURE

FIG. 1 FREEZING POINT APPARATUS FOR STYRENE

b) The change in ice-point reading of the thermometer obtained by comparison to its initial calibration against the standard thermometer; and

c) The correction for the emergent stem, which may always be taken as equal to −0·02°C.

A-4.6.2 *Precision* — shall be 0·05 mass percent of styrene over the range of 95 to 100 percent assuming the only impurity to be ethyl benzene.

A-4.7 Use of Bromobenzene for Standardization

A-4.7.1 The apparatus and procedure are to be checked with bromobenzene which has a freezing point close to that of styrene, under the following conditions:

a) When any doubt arises about the accuracy of results,

b) Whenever a new thermometer is used,

c) Whenever a new operator uses the apparatus, and

d) At least once every month with experienced operators and old thermometers.

A-4.7.2 Make sure that the thermomoter, stirrer, and the sample tube are clear and free from styrene. Freeze a portion of the standard sample of bromobenzene exactly as if running a test on styrene. Obtain the uncorrected freezing point by extrapolation of the freezing curve and apply the thermometer corrections.

The corrected freezing point should agree with the freezing point of the sample as originally determined with the standard thermometer within ± 0·02°C. If such agreement is not obtained, the ice-point of the thermometer should be redetermined as in **A-3**. If agreement between the corrected and predetermined freezing points is still not obtained, a source of error, particularly contamination of the sample with carbon dioxide, should be looked for in the method being followed. If necessary, as a final resort, the thermometer shall be recalibrated and checked by the National Physical Laboratory, New Delhi.

A-5. ASSAY

A-5.0 Outline of the Method — Styrene content is obtained from the freezing point of the material, assuming the total impurity as ethyl benzene. The GLC method in **A-12** shall be employed as referee method in case of dispute.

A-5.1 Apparatus

A-5.1.1 *Styrene Freezing Point Thermometer* — same as in **A-4.2.1**.

A-5.1.2 *Freezing Point Apparatus* — same as in **A-4.2.2**. A Dewar flask is preferable but an ordinary one-litre or large size, graduated cylinder may be used.

A-5.2 Procedure — Wash pieces of clear ice and crush them to a size not greater than 5 mm. Make a thick slurry by mixing a small amount of cooled distilled water with the crushed ice. Use a clean scoop rather than the hands for the manipulation of ice and the ice and water mixture. Fill the vessel with this mixture, tapping it down thoroughly. If the vessel is unsilvered, wrap it with a cloth. Support the thermometer in the ice and water mixture so that the zero degree mark is at the ice level. (A convenient support is made by putting a one-hole rubber stopper on top of the thermometer and clamping it). Place not more than two thermometers in the bath at one time, and be sure that the thermometers are suspended closer to the centre than to the outside of the cylinder. Note the time of immersion in the bath. Read the thermometer about 5 minutes after immersion in the bath. Take this reading as the ice-point, keeping the eye level with the top of the mercury column when taking the observations. The thermometer may be withdrawn slightly for readings but do not expose the enlargement which is below the auxiliary scale.

A-5.2.1 Take the freezing point reading as in **A-4.5**.

A-5.3 Calculation

$$\text{Assay, percent by mass} = 100 + 2{\cdot}47\,(\,t + 30{\cdot}63\,)$$

where

t = corrected freezing point of the material in °C from **A-4.6.1**.

NOTE — This equation is based on a value of —30.63°C for the freezing point of pure styrene saturated with air at one atmosphere pressure and a freezing point depression of 0·405°C for each mass percent of ethyl benzene as impurity.

Example

Uncorrected freezing point (from **A-4.5**)	−30·83°C
Calibration correction [**A-4.6.1** (a)]	−0·08°C
Change in ice-point reading [**A-4.6.1** (b)]	+0·01°C
Stem correction [**A-4.6.1** (c)]	−0·02°C
Corrected freezing point	−30·92°C

Styrene, percent by mass

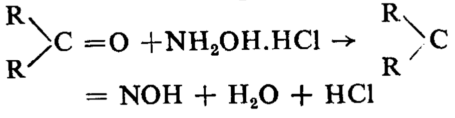
= 100+2·47 (− 30·92+30·63)

= 99·28

A-6. DETERMINATION OF ALDEHYDE CONTENT

A-6.0 Outline of the Method — A sample of styrene is added to an alcoholic solution of hydroxylamine hydrochloride. The aldehydes react with hydroxylamine hydrochloride (any ketones present will be partially included) according to the following equation:

$$RCHO + NH_2OH.\,HCl \rightarrow RCH = NOH + H_2O + HCl$$



$$R_2C = O + NH_2OH.HCl \rightarrow R_2C = NOH + H_2O + HCl$$

The hydrochloric acid liberated, which is equivalent to the aldehyde present in the sample, is titrated with standard alkali.

A-6.1 Reagents

A-6.1.1 *Hydroxylamine Hydrochloride Solution* — Dissolve 20 g of hydroxylamine hydrochloride (reagent grade) in one litre of methanol and neutralize to the red-yellow end-point of thymol blue indicator.

A-6.1.2 *Standard Sodium Hydroxide Solution* — 0·05 N.

A-6.1.3 *Hydrochloric Acid* —0·05 N.

A-6.1.4 *Methanol* — *see* IS : 517-1967*.

A-6.1.5 *Thymol Blue Indicator Solution*

A-6.2 Procedure — Pipette 25 ml of styrene sample into a 250-ml glass-stoppered Erlenmeyer flask containing 25 ml of methanol. Add 0·2 ml of thymol blue indicator and, if necessary, neutralize with 0·05 N sodium hydroxide or hydrochloric acid to the red-yellow end-point. Add 25 ml of the neutralized hydroxylamine hydrochloride solution and allow to stand for one hour,

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*Specification for methanol (methyl alcohol) (*first revision*).

shaking the flask occasionally. Titrate to the original red-yellow end-point with standard 0·05 N sodium hydroxide dispensed from a 10-ml burette, and record this value. Let it stand for another hour and again titrate any acid which may have been liberated.

A-6.3 Calculation

Aldehyde content (as benzaldehyde)

$$\text{percent by mass} = \frac{V \times N \times 10 \cdot 61}{25 \times 0 \cdot 9}$$

where

V = total volume in ml of sodium hydroxide required for both titrations, and

N = normality of standard sodium hydroxide solution used.

A-7. DETERMINATION OF PEROXIDES

A-7.0 Outline of the Method — A methanol solution of ferrous ammonium sulphate and potassium thiocyanate is acidified with dilute sulphuric acid and any ferric iron present is reduced by the careful addition of titanous chloride. A sample of styrene is then added and any peroxides present will oxidize the ferrous iron to ferric iron forming the red ferric thiocyanate colour. The ferric iron is titrated with titanous chloride that has been standardized freshly against ferric iron, and calculated as hydrogen peroxide. Any peroxide oxygen will be included.

A-7.1 Apparatus

A-7.1.1 *Burette* — A 50-ml long-tipped burette and, if desired, as assembly arranged for storing and using the titanous chloride solution under an inert atmosphere.

A-7.2 Reagents

A-7.2.1 *Titanous Chloride Solution* — Mix 10 ml of concentrated hydrochloric acid and 6·5 ml of 20 percent titanous chloride and heat on steambath in an inert atmosphere for ten minutes. Dilute to 500 ml with distilled water. This solution should be standardized once a day and preserved under carbon dioxide atmosphere.

A-7.2.2 *Standard Iron Solution* — Dissolve 35·11 g of crystallized ferrous ammonium sulphate [$FeSO_4.(NH_4)_2SO_4.6H_2O$] in 400 ml of distilled water. Add 25 ml of concentrated sulphuric acid, warm the solution, and add potassium permanganate solution until the iron is completely oxidized. Dilute to 1 litre with distilled water. One mililitre contains 0·005 g of ferric-iron.

A-7.2.3 *Ferrous Ammonium Sulphate* — Dissolve 39·2 g of ferrous ammonium sulphate in 800 ml of distilled water. Add 50 ml of concentrated sulphuric acid and dilute to one litre with distilled water.

A-7.2.4 *Potassium Thiocyanate* — 10 percent aqueous solution.

A-7.2.5 *Sulphuric Acid* — 1 : 1 aqueous solution.

A-7.2.6 *Hydrochloric Acid* — conforming to IS : 265-1976*.

A-7.2.7 *Methanol, Absolute*

A-7.2.8 *Standard Titanous Chloride Solution (0.2 N)* — Mix 150 ml of 20 percent titanous chloride solution with 100 ml of concentrated hydrochloric acid. Dilute to one litre with distilled water. Mix the solution thoroughly by passing a current of oxygen-free inert gas through it. If necessary, filter the solution through a filter paper† until the filtrate is completely clear. Agitate the solution thoroughly with the inert gas for a few minutes and store the solution in the amber storage bottle with inert atmosphere. Standardize with ferric alum solution.

NOTE — For the preparation of 0·02 N standard Solution from 20 percent titanous chloride solution, mix 15 ml of the 20 percent titanous chloride solution with 10 ml of 38 percent hydrochloric acid and proceed as above.

A-7.3 Standardization of Titanous Chloride Solution — Pipette 10 ml of the standard iron solution into a 250-ml wide-mouth conical flask containing 50 ml of water, 15 ml of concentrated hydrochloric acid and 10 ml of 10 percent potassium thiocyanate. Add a small lump of solid carbon dioxide to the flask to maintain an inert atmosphere over the solution at all times. Keep the tip of the burette low in the flask and titrate with the titanous chloride solution until the pink colour of the ferric thiocyanate just disappears.

A-7.3.1 *Calculation* — Hydrogen peroxide factor of titanous chloride, that is, mass in g of hydrogen peroxide equivalent to 1 ml of the titanous chloride solution, is calculated as:

$$\text{Factor} = \frac{0 \cdot 05 \times 17 \cdot 01}{T_s \times 55 \cdot 85}$$

where

T_s = volume in ml of titanous chloride required for the titration of 0·05 g of ferric iron contained in 10 ml of the standard iron solution.

A-7.4 Procedure — Pipette 10 ml of approximately 0·1 N ferrous ammonium sulphate into a 250-ml wide-mouth conical flask containing 100 ml of methanol, 5 ml of 10 percent potassium thiocyanate, and 2 ml of 1 : 1 sulphuric acid. Add a small lump of solid carbon dioxide to the flask, mix by swirling, cover with a watch-glass and allow to stand for 15 minutes. Carefully add the

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*Specification for hydrochloric acid (*second revision*).

†Whatman No. 41 paper or equivalent is suitable.

titanous chloride solution, while keeping the tip of the burette low in the flask, until the pink colour just disappears. This amount of titanous chloride used is not required in the calculation. Pipette 25 ml of the sample of styrene into the above adjusted solution, mix by swirling, cover with a watch-glass, and allow to stand for 2 hours.

Titrate with the standard titanous chloride solution until the pink colour of the ferric thiocyanate just disappears.

A-7.5 Calculation

Hydrogen peroxide, percent by mass

$$= \frac{T \times \text{Factor} \times 100}{25 \times 0{\cdot}9}$$

where

T = volume in ml of standard titanous chloride required for the ferric iron produced by the peroxides in the sample, and

Factor = mass in g of hydrogen peroxide equivalent to 1 ml of the titanous chloride (*see* **7.3.1**).

A-8. DETERMINATION OF CHLORIDES

A-8.0 Outline of the Method — A sample of the material is mixed with alcohol to aid combustion and is burnt in chloride-free air. The products of combustion are bubbled through sodium hydroxide solution which retains the chlorides as sodium chloride. The acidity and the volume of the absorbing solution are adjusted, the chloride precipitated as silver chloride, and the turbidity of the solution determined by means of a suitable turbidimeter.

A-8.1 Apparatus

A-8.1.1 *Air Purification* — The air is passed first through a 2·5 × 30 cm straight drying tube filled with coarse calcium chloride, then through a 2·5 × 85-cm quartz or Vycor glass tube packed with 50 cm of coarse cupric oxide (CuO), 15 cm of asbestos impregnatad with lead chromate, and a 15 cm roll of copper gauze. The cupric oxide portion of the tube is heated to approximately 700°C in an electrical resistance furnace. The air is finally passed through a large manifold reservoir, the size of which depends on the number of lamps used — the larger the reservoir, the more uniform is the flow of air to all lamps and fewer burner manipulations are required.

A-8.1.2 *Lamp* — The lamp consists of a burner, chimney, absorber, and condenser and the inner tube of the lamp uses 5 full strands of the cotton.

A-8.1.3 *Alcohol Lamp* — for igniting the burner.

A-8.1.4 *Suitable Turbidimeter* — preferably. Hellige Model A turbidimeter used for water analysis. Any other turbidimeter may also be used which shall be accurate enough as to give readings corresponding to Hellige Model A turbidimeter.

A-8.2 Reagents

A-8.2.1 *Sodium Hydroxide Absorbing Solution* — Dissolve 10 g of sodium hydroxide in 1 litre of distilled water.

A-8.2.2 *Nitric Acid* — 1 N solution.

A-8.2.3 *Phenolphthalein Indicator* — *see* IS : 2263-1979*.

A-8.2.4 *Ethanol* — Conforming to IS : 321-1964†.

A-8.2.5 *Nitric Acid-Silver Nitrate Reagent* — Prepare a solution approximately 0·2 N of nitric acid and 0·01 N of silver nitrate by diluting 12·5 ml of concentrated nitric acid to 1 litre and adding 1·70 g of silver nitrate (*see* IS : 2214-1977‡).

A-8.2.6 *Standard Chloride Solution* — Dilute to 1 litre with distilled water 10 ml of a solution containing 4·12 g of reagent grade sodium chloride per litre. This solution contains 25 mg of chloride per litre. One ml diluted to 50 ml, as in the procedure, gives a solution corresponding to 0·5 ppm of chloride.

A-8.3 Procedure — Place 30 ml of the sodium hydroxide absorbing solution in the absorber. Adjust the cotton lampwick until it is slightly above the rim of the burner, and with the burner inserted in the chimney, adjust the flow of air to the approximate velocity necessary for burning. Remove the burner and add about 25 ml of a mixture consisting of 15 ml of styrene sample in 40 ml of absolute alcohol. Weigh the lamp plus all the sample. Ignite the wick with the flame of an alcohol lamp and re-insert the burner in the chimney. Adjust the air flow to give a clear and smokeless flame.

A-8.3.1 When about 20 ml of the mixture has been burnt, reweigh the lamp and the residual unburnt portion of the sample, and transfer the absorbing solution to a 400-ml beaker. Rinse the apparatus several times with distilled water. The final volume should be about 200 ml. Evaporate to about 10 ml on a hot-plate in a fume hood free from chloride fumes.

A-8.3.2 Filter the evaporated solution through a filter paper§ into a 50 ml volumetric flask,

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*Methods of preparation of indicator solutions (*first revision*).

†Specification for absolute alcohol (*revised*).

‡Specification for silver nitrate, pure and analytical reagent (*first revision*).

§Whatman No. 42 paper or equivalent is suitable.

washing the beaker several times with distilled water. Make the filtrate neutral to phenolphthalein with 1 N nitric acid. The resulting volume should approximate 200 ml. Add 20 ml of ethanol. The bicarbonate present may cause the solution to become turbid at this point; if so, carefully add 1 N nitric acid dropwise until all of this turbidity is dispelled. Add dropwise 5 ml of the nitric acid-silver nitrate reagent, then make up to the mark with ethanol and shake. Place the flask in a water-bath at 40°C for 35 minutes, then cool the sample rapidly to room temperature.

A-8.3.3 Transfer the contents of the flask to a 20 mm optical cell of the turbidimeter. Stir in an additional 10 ml of ethanol and determine the turbidity. Compare the readings against the standard curve to find parts per million of chloride. Concentrations of greater than 5 ppm chloride should be determined on aliquots of the sample.

A-8.3.4 Determine a blank test on the apparatus and air stream, following the same procedure described above but omitting the sample. Burn the blank for the average length of the time required for the sample.

A-8.3.5 Prepare the standard chloride calibration curve by measuring out aliquot portions of the standard chloride solution and analyzing by the above procedure.

A-8.4 Calculation

Chloride (as Cl), percent by mass

$$= \frac{(A-B) \times 0{\cdot}1}{S}$$

where A = mg chloride in the sample,
B = mg chloride in the blank, and
S = g of sample burnt.

A-9. DETERMINATION OF POLYMER CONTENT

A-9.0 Outline of the Method — This method is intended to determine trace quantities of polymer in styrene. When the styrene is added to dry methanol, the polymer precipitates and the resultant turbidity is quantitatively estimated by means of a turbidimeter or colorimeter.

A-9.1 Apparatus

A-9.1.1 *Turbidimeter or Colorimeter*

A-9.1.2 *Pipette* — 25-ml and 10-ml.

A-9.2 Reagents

A-9.2.1 *Methanol, Analytical Reagent Grade*

A-9.2.2 *Standard Polymer Solution* — Fill a 20 × 175 mm test tube one third full of uninhibited styrene and place it in an oven at 150°C for about 60 hours. After the styrene has polymerized, allow the polymer to age at room temperature for three or four days.

Dissolve 0·450 0 g of the styrene polymer in freshly distilled polymer-free inhibited styrene in a 100-ml volumetric flask. When the polymer is dissolved, dilute to volume with additional polymer-free styrene. This solution contains 5 000 ppm (*m*/*m*) polymer.

Dilute further to yield solutions containing 5, 10, 15, 20, ... 50 ppm (*m*/*m*) of polymer.

A-9.3 Procedure — Take 20 ml of dry methanol in a 100-ml glass stoppered flask, add 20 ml of standard polymer solution and shake vigorously for 30 seconds and allow it to stand for five minutes. Read the percent transmission on the colorimeter using dry methanol and polymer free styrene as blank at 470 mμ.

Draw a graph of percent transmission against ppm polymer. To estimate polymer in the given sample of styrene, follow the above procedure and read off ppm polymer against percent transmission observed, from the standard graph prepared.

NOTE — If the precipitate is in excess, dilute the original sample to a suitable range with pure styrene, toluene or benzene and follow the above procedure. Then apply the dilution factor to the result to compute the polymer content in the original sample.

A-10. DETERMINATION OF SOLUBILITY OF POLYMER

A-10.0 Outline of the Method — This method is intended as a qualitative test to detect cross-linking material, such as divinylbenzene, in the styrene. A portion of the styrene sample is polymerized by means of benzoyl peroxide. A sample of the polymer is mixed with benzene. Any undissolved polymer indicates the presence of cross-linking impurities. Polystyrene is readily soluble in benzene whereas cross-linking impurities in the styrene produce a polymer that does not readily dissolve in benzene.

A-10.1 Reagents

A-10.1.1 *Benzoyl Peroxide, Reagent Grade*

A-10.1.2 *Benzene, Reagent Grade* — Conforming to IS : 1840-1961*.

A-10.2 Procedure — Place 0·2 g of the benzoyl peroxide in a 152 × 16 mm soft-glass test tube and add 5 g, or 5·5 ml, of the styrene sample under investigation. Heat the solution on a steam-bath for 16 hours, leaving the test tube unstoppered. Cool the test tube and break away the glass. Grind the polymer in a mortar and place 1 g in a 50-ml beaker with 10 ml of toluene. Allow to stand for 2 hours. The styrene polymer should be completely dissolved at this time. Any insoluble material indicates the presence of cross-linking impurities.

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*Specification for benzene, reagent grade.

NOTE — Care shall be taken in conducting the polymerization as the reaction may become extremely violent.

A-11. DETERMINATION OF INHIBITOR CONTENT

A-11.0 Outline of the Method — This method determines the amount of 4-*t*-butyl-1, 2 dihydroxy benzene, TBC added to styrene to prevent polymerization while in transit and storage. It determines only the active inhibitor.

A-11.0.1 Catechols in alkaline solution readily absorb oxygen, with the formation of highly coloured compounds. TBC gives a pink to blood-red colouration, that is, proportional to the amount of catechol present. The colour is apparently caused by the oxidation of the dihydroxy molecule to the quinone. This is measured using Nessler tube or any suitable colorimeter.

A-11.1 Apparatus

A-11.1.1 *Colorimeter*

A-11.1.2 *Pipette* — 50-ml.

A-11.1.3 *Separating Funnel* — 125-ml.

A-11.2 Reagents

A-11.2.1 *Sodium Hydroxide Solution* — 1 N.

A-11.2.2 *Standard Inhibitor Solution* — In 1 000 ml of uninhibited stryrene prepared by washing 4 volumes of styrene three times with one volume of 10 percent aqueous solution of sodium hydroxide, dissolve 0·113 g of TBC. This solution shall contain 125 ppm of TBC.

A-11.2.3 *Styrene Uninhibited* — Wash 4 volumes of styrene three times with one volume of 10 percent reagent grade sodium hydroxide. Referigerate to prevent polymerization.

A-11.3 Procedure — Prepare standard solutions containing 0, 5, 10, 15 ... 100 ppm TBC by adding 0, 2, 4, 6 ··· 40 ml of standard TBC solution, respectively to 125 ml separating funnels. Add sufficient inhibitor-free styrene to make a total volume of 50 ml.

To each separating funnel, add 50 ml of 1·0 N caustic soda solution and shake for 30 to 60 seconds and allow the two layers to separate. Repeat the shaking of the separating funnel three to five times and finally allow clear separation to take place. Filter the aqueous layer through a coarse filter paper into a 100-ml Nessler tube. Then measure the percent transmission on the colorimeter at 470 mμ observing all the necessary precautions and instructions of the instrument, using 1·0 N caustic soda solution as blank.

Draw a graph of percent transmission against ppm, TBC.

A-11.3.1 Take 50 ml of styrene in a separating funnel. Add 50 ml of 1·0 N caustic soda solution and shake for 30 to 60 seconds. Allow the two layers to separate. Repeat the shaking of the separating funnel three to five times and finally allow clear separation to take place. Filter the aqueous layer through a coarse filter paper into a 100-ml glass tube. Then measure the percent transmission on the colorimeter at 470 mμ observing all the necessary precautions and instructions of the instrument using 1·0 N caustic soda solution as blank.

A-11.3.2 Compare the reading with the standard graph and read the amount of TBC in the sample.

NOTE — If the concentration of TBC goes out of the range of the standard curve, take a smaller sample and find out the percent transmission observing the above procedure. The result must be corrected by the factor (*F*) to give the actual concentration of TBC ,

$$\text{Factor } (F) = \frac{50}{\text{sample size in ml}}$$

A-12. ASSAY AND DETERMINATION OF IMPURITIES

A-12.0 General — The chromatographic conditions given here are for guidance only. Variations in conditions to arrive at the typical chromatographic separation shown in Fig. 2 are possible.

A-12.1 Outline of the Method — A sample of the material is injected into the gas chromatograph from where it is carried by the carrier gas from one end of the column to the other end. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after the other and are detected by suitable means whose response is related to the amount of specific component leaving the column.

A-12.2 Apparatus

A-12.2.1 Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. A typical chromatogram using such a chromatograph is shown in Fig. 2 which may be obtained with the following chromatographic conditions:

Column	: Copper column; length × ID : 1 100 × 4 mm. Filled with 3 percent Apiezon L with 0·25 percent tertiary butyl catechol on 30-60 mesh chromosorb (P).

Carrier gas : Hydrogen

Detector : Thermal conductivity

Conditions : Column temperature — initially 170°C and temperature programming at 5°C/min from start.
Injection temperature, 150°C
Detector temperature, 220°C
Flow rate, 45·0 ml/min

Sample size : 2μl

A-12.3 Procedure — Conduct the flow of the carrier gas and inject sample at injection port where it is vaporized and well mixed with carrier gas. This is led into the chromatographic column wherein vapourized constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. For this separation to be efficient, it is necessary that the column is maintained at the temperature suggested throughout the time required for the resolution of the constituents. As the sample enters the detector, it gives the single corresponding to the amount of particular constituents leaving the column. The detector signal, on transmission to the recorder, plots the chart. From the areas under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

A-12.4 Calculation

A-12.4.1 Calculate the peak areas of individual constituents pertaining to ethyl benzene and alpha-methylstyrene on the chromatogram of the material. The concentration of these impurities may be obtained on the basis of peak area on chromatogram obtained with known amount of pure ethyl benzene and alpha-methylstyrene using the same apparatus under identical conditions.

A-12.4.2 Assay, percent by mass, is determined by subtracting the sum of percent impurities from 100.

A-12.5 Results

A-12.5.1 The typical chromatogram is shown in Fig. 2.

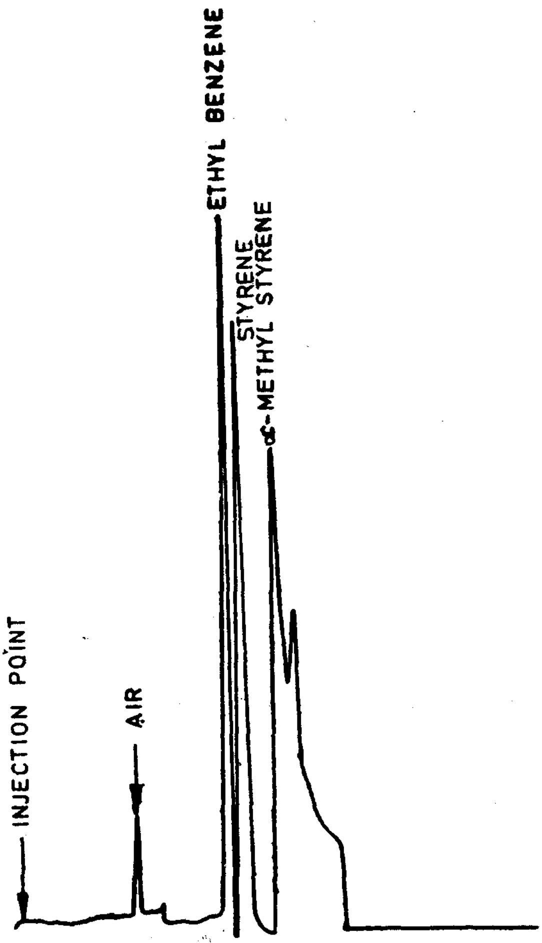


FIG. 2 A TYPICAL CHROMATOGRAM FOR STYRENE

APPENDIX B

(*Clause* 5.1)

SAMPLING OF STYRENE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry and shall be made of low or reduced spark generating material.

B-1.3 The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or both, or by rolling, so as to bring all portions into uniform distribution.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass containers preferably of amber or blue colour.

B-1.6 The sample containers shall be of such a size that they are almost, but not completely, filled by the sample.

B-1.7 Rubber stoppers, or composition cork shall not be used for closing the sample bottles.

B-1.8 Sealing wax or other plastic sealing material, if used, shall be applied in such a way that it does not contaminate the sample when the bottles are opened.

B-1.9 Each sample container shall be protected by covers of oilproof paper, metal foil, viscose or other suitable impervious material over the stopper to keep the moisture and dust away from the mouth of the bottle and to protect it while being handled.

B-1.10 Each sample container shall be sealed air-tight with a suitable stopper after filling and marked with full details of sampling, such as the grade of styrene, the date of sampling, the year of manufacture of the material, the batch number, the name of the sample, etc. Particular care shall be taken to ensure that sealing methods do not contaminate the sample.

B-2. SAMPLING INSTRUMENT

B-2.0 The following forms of sampling instruments may be used:

a) Sampling bottle or can for taking samples from various depths in large tanks, and

b) Sampling tube.

B-2.1 Sampling Bottle or Can — It consists of a weighed bottle or metal container with removable stopper or top, to which is attacked a light chain (*see* Fig. 3). The bottle or can is fastened to a suitable pole. For taking a sample, it is lowered in the tank to the required depth, and the stopper or top is removed by means of the chain for filling the container.

B-2.2 Sampling Tube — It is made of metal or thick glass and is about 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 4). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and is finally closed and withdrawn.

B-2.2.1 For small containers, the size of the sampling tube may be altered suitably.

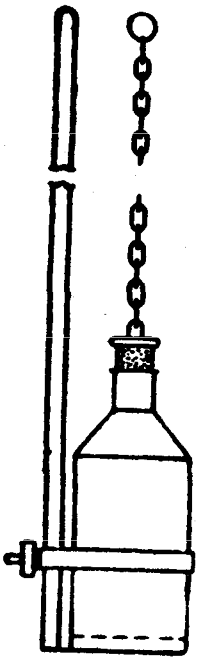
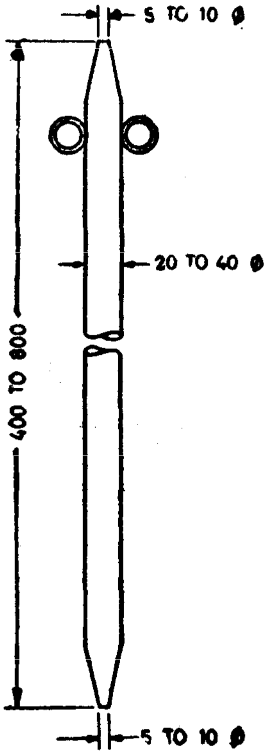


FIG. 3 SAMPLING BOTTLE OR CAN



All dimensions in millimetres.

FIG. 4 SAMPLING TUBE

B-3. SCALE OF SAMPLING

B-3.1 Lot — In any consignment, all the containers of same size and grade drawn from the same batch of manufacture shall constitute a lot.

B-3.2 Tests shall be conducted on each lot of each grade of styrene, separately for ascertaining its conformity to the requirements of this specification. The number of containers to be chosen at random from the lot for this purpose shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 SCALE OF SAMPLING

Lot Size N	No of Containers to be Selected n
(1)	(2)
Up to 50	3
51 to 100	4
101 to 150	5
151 and above	7

Note — In case of very small lots where the selection of three containers may be uneconomical, the number of containers to be selected and the method of judging the conformity of the lot to the requirements of specification shall be as agreed to between the purchaser and the supplier.

B-3.3 The containers shall be selected at random from the lot. In order to ensure the randomness of selection, procedure given in IS : 4905-1968* may be followed.

B-4. PREPARATION OF TEST SAMPLES

B-4.1 From each of the containers selected according to **B-3.3**, a small representative portion of the material not less than 600 ml, shall be drawn with the help of suitable sampling instrument (*see* **B-2**).

B-4.2 Out of these portions, a small but equal quantity of material shall be taken and mixed thoroughly to form a composite sample of about 600 ml. The composite test sample shall be divided into three equal parts one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-4.3 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *n* containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-4.4 All the individual and composite samples shall be transferred to separate containers and shall be sealed and marked with full identification particulars given under **B-1.10**.

B-4.5 The referee test samples consisting of a composite sample and a set of *n* individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of any dispute between the two.

B-5. NUMBER OF TESTS

B-5.1 Tests for determination of freezing point and assay shall be conducted on each of the individual samples (*see* **B-4.3**).

B-5.2 Tests for determination of remaining characteristics shall be conducted on the composite sample (*see* **B-4.2**).

B-6. CRITERIA FOR CONFORMITY

B-6.1 The lot shall be declared as conforming to the requirements of this specification if the following conditions are satisfied:

a) *Individual Samples* — For individual test results of freezing point and assay, the average ($\bar{X}$) and range (R) shall be calculated and shall satisfy the expression $\bar{X} - 0{\cdot}6\,R \geqslant$ the minimum specified value.

b) *Composite Sample* — The composite sample shall conform to the remaining requirements given in **3.1** and Table 1.

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*Methods of random sampling.

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